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Semi-Solid Metal Processing – A Processing Method Under Low Flow Loads

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Received Date: 6th June 2006 Accepted Date: 19th December 2006

ABSTRACT

Semi-solid metal processing, also known as thixoforming, is a forming process that shapes metal components in their semisolid state. For this to be possible, it is preferable for an alloy to have an appreciable melting range and before forming the microstructure must consist of solid metal spheroids in a liquid matrix. There is strong interest in thixoforming high temperature materials such as tool steels. The established solid state processing approach to obtaining a non-dendritic microstructure is through recrystallisation processes of RAP (Recrystallisation And Partial melting) and SIMA (Strain Induced Melt Activated). The first involves cold or warm working below the recrystallisation temperature followed by heating above the solidus of the material, while the latter involves hot working between recrystallisation and solidus temperatures, followed by cold working so as to produce a critical level of strain, before the material is reheated above the solidus. Here, a commercially produced M2 tool steel alloy, i.e. being hot worked, tempered and annealed, was subjected to a direct partial remelting experiment in a protective atmosphere from room temperature with no prior additional cold working. The microstructures revealed equiaxed solid grains (in liquid matrix) of average sizes between 29 and 31 μm when reheated to above the solidus of 1320-1360°C. Subsequent thixoforming of the material in the form of a slug of 36 mm diameter and 46 mm height into a simple finger of 66 mm long, 43 mm wide and 7 mm thick at 1360°C showed that the slurry flowed thixotropically under very low flow load of typically less than about 2 kN.

Keywords: Semi-solid metal processing, M2 tool steel, direct partial remelting

ABSTRAK

Pemprosesan logam separa-pejal, juga dikenali sebagai pembentukan-tikso, adalah satu proses pembentukan komponen logam yang dilakukan apabila bahan berkenaan berada dalam keadaan separa pepejal. Proses ini cenderung kepada logam yang mempunyai julat suhu lebur yang besar di samping *struktur fasa pepejal yang berbentuk sfera. Kajian yang melibatkan logam yang mempunyai takat lebur yang tinggi seperti keluli alat semakin mendapat perhatian penyelidik. Antara kaedah dikenal pasti untuk mendapatkan struktur fasa pepejal berbentuk sfera adalah kaedah RAP (Recrystallisation and Partial Melting) dan SIMA (Strain Induced Melt Activated). RAP melibatkan kerja sejuk di bawah suhu penghabluran semula diikuti dengan pemanasan melebihi suhu solidus logam terbabit. SIMA pula melibatkan kerja panas di antara suhu penghabluran semula dan suhu solidus bahan, diikuti dengan proses kerja sejuk untuk menghasilkan nilai terikan kritikal. Kajian ini melibatkan keluli alat M2 yang diperolehi secara komersial (iaitu telah dikenakan proses kerja panas, pembajaan dan penyepuhlindapan) dan dikenakan ujian peleburan-separa-langsung (direct partial remelting) tanpa melalui proses pembentukan sfera pepejal seperti dua kaedah di atas. Sebaliknya ia membabitkan kaedah pemanasan secara langsung ke zon separa pepejal bahan tanpa terlebih dahulu dikenakan proses kerja sejuk. Mikrostruktur menunjukkan ira pepejal sama dimensi dengan saiz purata antara 29 hingga 31 μm telah diperolehi apabila bahan dipanaskan ke julat suhu 1320-1360°C. Pembentukan-tikso yang dilakukan ke atas bilet bersaiz 36 mm diameter dan 46 mm tinggi untuk menghasilkan komponen ujian berbentuk jejari berdimensi 66 mm panjang, 43 mm lebar dan 7 mm tebal pada suhu 1360°C telah menunjukkan bahawa buburan separa-pejal telah mengalir secara tiksotropik dengan daya pengaliran yang serendah 2 kN semasa proses pembentukan dijalankan.*

Kata kunci: Pemprosesan logam separa-pejal, keluli alat M2, peleburan-separa-langsung

INTRODUCTION

Thixoforming is a forming process that shapes metal components in their semisolid state. Prior to forming, the microstructure of the alloy consists preferably of solid metal spheroids in a liquid matrix. The semi-solid slurry gives a peculiar flow behaviour, in that, its viscosity is dependent on shear rate and time. If an alloy in this state is sheared, this will result in a fall in its viscosity and it will flow like a liquid, but if allowed to stand it will stiffen and becoming more viscous. This non-Newtonian behaviour, was first discovered by Spencer et al. (1972) and has since led to extensive work on the thixotropy of alloy slurries. Reviews of the development of semi-solid processing have been given by Flemings (1991), Kirkwood (1994) and Fan (2002). The established solid state processing approach to obtaining a non-dendritic microstructure is through recrystallisation processes of RAP (Recrystallisation And Partial melting) and SIMA (Strain Induced Melt Activated). The first involves cold or warm working below the recrystallisation temperature followed by heating above the solidus of the material, while the latter involves hot working between recrystallisation and solidus temperatures, followed by cold working so as to produce a critical level of strain, before the material is reheated above the solidus. Here, a commercially produced M2 tool steel alloy, i.e. being hot worked (by GFM forging, from the German words for *Gesellschaft für Fertigungstechnik und Mashinenbau*), tempered and annealed, was directly reheated in a protective atmosphere from room temperature to above its solidus with no prior additional cold working. GFM forging is based on the radial forging principle whereby a long workpiece is hammered by four forging tools.

One major attraction of thixoforming high temperature materials (e.g. steels), is the low forging force involved during thixoforming as compared to that in conventional forgings (Nohn et al. 2000). This means that more intricate and complex shapes can be formed faster with some reduction in forming steps and with near net shaping capabilities (Nohn et al. 2000, Kapranos et al. 2000 & Omar et al. 2004 & 2005). Other major advantages include prolonged die life due to less thermal shock (forging below liquidus as against castings), weight savings in components with less porosity than conventionally, plus improved usage of feedstock materials because of improved designs.

This paper describes the microstructural development of the GFM M2 tool steel when directly reheated into its semi-solid zone from as-annealed condition. The phase changes are also described from a differential thermal analysis heating curve and a pseudobinary equilibrium phase diagram. Lastly, thixoforming of the M2 demonstrator parts are discussed using the results of their load-displacement signals and the resulting microstructures.

EXPERIMENTAL PROCEDURES

Material

The M2 high speed steel used was produced by GFM hot forging at the temperature of approximately 1150°C. It was then tempered at 650-750°C for about 4 hours, and subsequently annealed for 8-10 hours at 860°C before being furnace cooled to ambient temperature. All the heat treatment was carried out in a controlled atmosphere (in argon) to prevent oxidation and de-carburisation. The chemical composition of the starting material is given in Table 1.

TABLE 1. Chemical composition of the M2 tool steel compared to the nominal.

M2	Chemical composition (wt.%)*								
	C	W	Mo	Cr	V	Co	Si	Mn	Ni
Nominal	0.85	6	5	4	2	-	-	-	-
Chemical analysis	0.87	6.20	4.68	3.98	1.72	0.48	0.07	0.04	0.02

* balance is Fe

Differential Thermal Analysis (DTA)

DTA analysis was carried out primarily to estimate the solidus, liquidus and liquid fractions within the semi-solid zone of the supplied material. The alloy was cut into small pieces of 70-90mg weight for DTA tests using a Perkin Elmer Differential Thermal Analyser DTA7. Alumina of about 100mg weight was used as the reference material. The heating rate employed was 20°C per minute. The heating was carried out in an argon atmosphere to prevent oxidation. From the heating curve, phase transformation reactions are also observed.

Direct partial remelting experiments

Partial remelting was carried out using a vertical high temperature quench Carbolite tube furnace, capable of reaching a maximum temperature of 1500°C. The as-received billet was cut into coupons of approximately 5x10x12mm. An K-type thermocouple was placed inside a hole located on the 5x10mm surface of the coupon (5-6mm deep), to ensure that the sample had reached the predefined quenching temperature, and hung inside the furnace. The as-received samples were subjected to different holding temperatures and times. They were quenched in a salt bath after the predetermined experimental parameters to freeze the structures. To ensure that the sample is cooled rapidly, its dimensions must be kept to a minimum while at the same time having enough strength in the mushy state to provide the presence of thick walls around the thermocouple inside the thermocouple hole. A coupon thickness of 5mm was found suitable for the purpose. The selected temperatures were 1320, 1340 and 1360°C at various holding times of 0, 4 and 8 minutes. The heating was carried out in an argon atmosphere to reduce oxidation.

Image analysis

The microstructural characterisation was carried out using KS-400 Imaging System Release 3.0 software connected to a Reichert-Jung Polyvar MET optical microscope and Jeol JSM 6400 scanning electron microspcope. The grain or cell size and the volume fraction of liquid were respectively measured adopting the Mean Lineal Intercept method as outlined in ASTM E112-96 standard (1996) and by the Systematic Manual Point Count as outline in ASTM E562-99 standard (1999). All samples were etched using 5% Nital (5ml HNO_3 + 95ml methanol or ethanol).

Thixoforming

Thixoforming was carried out using the thixoforming press at the University of Sheffield, Sheffield, United Kingdom (details of the press is given in Kapranos et al. 1993) and a slug size of 36mm diameter by 46mm high. The press has the capacity of producing a 100kN load during forging and is able to transfer a slug of material to be injected into a die at a maximum velocity of 1000mm/s. It is controlled by a computer equipped with a Servotest DCS 2000 digital control system. The data collection acquisition rate is up to 2kHz. Load-displacement signals during forging were obtained from a pressure transducer sensing hydraulic fluid pressures exerted by the ram. The signals obtained are collected by the computer controller and easily transferred into Microsoft Windows Excel software for analysis.

Graphite was selected as the die material based on work by Kapranos et al.on thixoforming of some high temperature materials (Kapranos et al. 1993). The dies were housed in a split steel die box and clamped at the top of the press. They produced parts of 66mm long, 43mm wide and 7.5 mm thick. The slug was heated in an argon gas environment to reduce oxidation. The heating was by means of an induction furnace system having a current frequency of 1000Hz, with the induction coil having a diameter of 130mm. The formings were carried out at 500mm/s die filling velocity (ram speed), 10 seconds dwell time and final load of 75kN at processing temperatures of 1340 and 1360°C. The top and bottom surfaces of the slug were respectively insulated with a layer of insulating Kaowool pad of approximately 1mm thick. Kapranos et al. (1996) had shown that this procedure had reduced heat losses from the outer surface with the top corners being the coldest spots. As a result, a much more uniform temperature distribution is achieved in the slug during heating.

RESULTS AND DISCUSSION

Starting material

Figures 1 (a) and (b) are the scanning electron micrographs under back-scattered electron imaging mode of the as-received M2 tool steel. Figure 1(a) shows excess carbide particles in ferrite matrix that are contained in bands parallel to the working direction along the as-received billet. The structure represents a typically good manufacturing practice of most commercially produced high speed steels (Robert et al. 1971). Figure 1(b) shows two different types of carbides present in the as-received M2, the whitish and grayish carbides. Energy Dispersive Spectroscopy (EDS) analyses showed that the former is rich in Tungsten and Molybdenum (an M_6C type carbide), while the latter is a Vanadium-Tungsten-Molybdenum rich MC type carbide. The absence of $M_{23}C_6$ Chromium rich carbide can be explained by the fact that this type of carbide is dissolved at the high commercial hardening temperature (~1200°C) normally carried out on M2; and since the as-received M2 in this work was hot forged at 1150°C, the absence of $M_{23}C_6$ is expected.

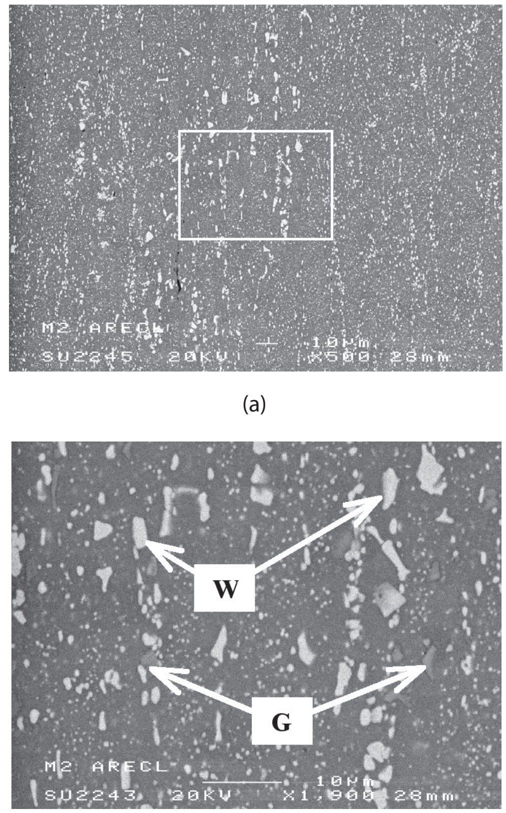

FIGURE 1. SEM (backscattered) micrographs of as-received GFM M2 showing carbide segregations caused by forging, (a), and (b) is the area as marked in (a) showing whitish (W) and grayish (G) carbides. (Longitudinal)

Phases and microstructures within semi-solid zone

Prior to the partial remelting experiments, DTA analysis was carried out to estimate the solidus, liquidus and liquid fractions within the semi-solid zone of the as-received M2. The DTA heating curve, together with its corresponding liquid distribution, is shown in Figure 2. The solidus is estimated at 1235°C and the liquidus at 1458°C, while 20% and 50% liquid correspond to 1330°C and 1380°C respectively. Note that thixoforming is normally carried out at fraction liquid between 20-50% (Burke 1998, Chayong et al. 2000 & Omar et al. 2005).

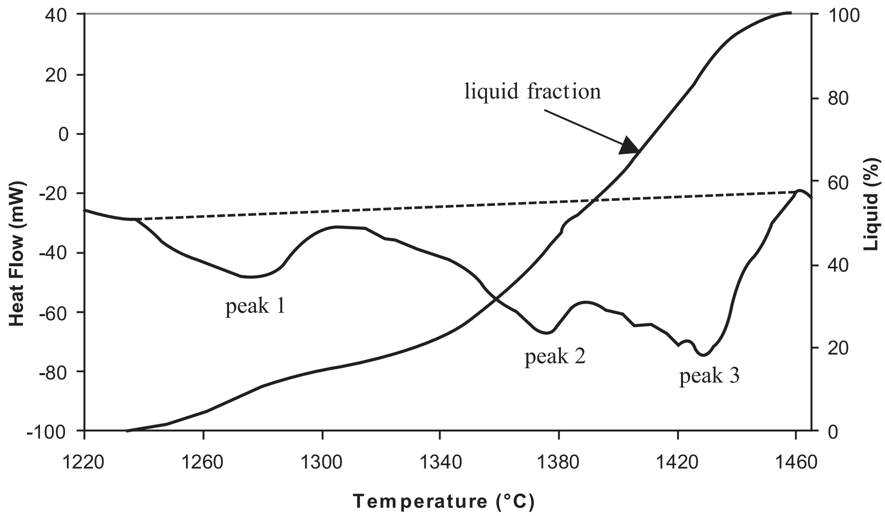

FIGURE 2. DTA (heating at 20°C/min) and the corresponding liquid fraction profiles of M2. Dotted line indicates baseline, constructed using a 'common tangent' procedure.

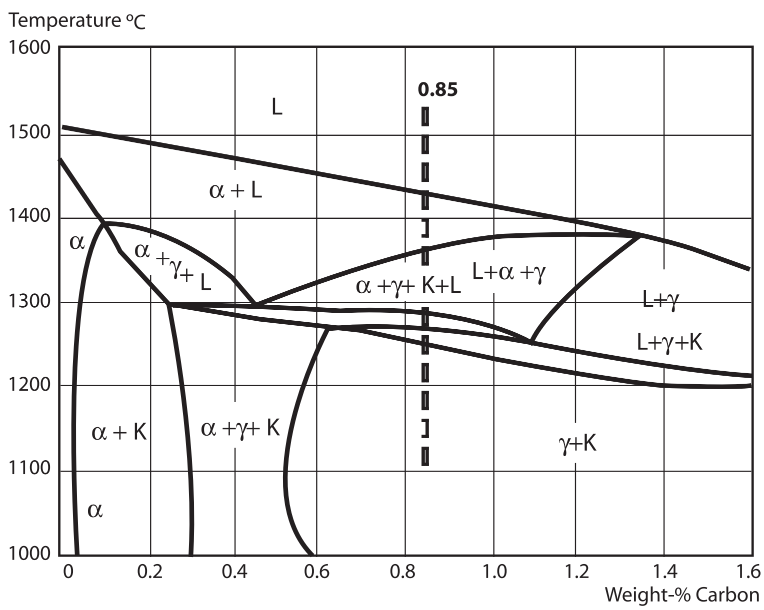

FIGURE 3. Pseudobinary equilibrium phase diagram for high speed steels with approximately 4%Cr, 5%Mo, 6%W and 2%V (weight percent). L – liquid, α- ferrite, γ - austenite and K – carbides. (Jerkontoret 1977)

The estimation of the solidus and liquidus temperatures and also the liquid fraction at various temperatures in the semi-solid zone, is helpful in determining the temperatures for the isothermal re-heating tests in the semi-solid zone. The DTA curve exhibits three major endothermic peaks which are caused by transformation reactions. As a guideline, the phase reactions can be specified using a pseudobinary equilibrium phase diagram (at 0.85% carbon) as shown in Figure 3 (Jerkontoret 1977). Table 2 shows the expected phases and their respective stability ranges. The carbide denoted as C is expected to be a mixture of tungsten and molybdenum rich carbides of M_6C and M_2C. The first and second endothermic peaks of the DTA heating curve (Figure 2) are associated with the end of the C and MC carbides dissolution respectively, while the third is the end of austenite dissolution.

TABLE 2. Expected phases within the semi-solid state of GFM M2 (L - liquid, A – austenite, δ - delta ferrite, C and MC - carbides)

Temperature range, °C	Phase	Liquid, %
1235 – 1275	L + C + MC + A	0 – 37
1275 – 1376	L + MC + A	37 – 44
1376 – 1427	L + A + δ	44 – 85
1427 – 1458	L + δ	85 – 100
>1458	L	100

The quenched coupons from the direct remelting experiments could be used to assess further the liquid fraction and grain size against temperature and holding time. Figure 4 shows the typical structures at 1340°C and 1360°C at zero holding time. The structures show fine equiaxed solid grains in liquid matrix, which is suitable for thixoforming process. If enough wetting is achieved along grain boundaries, initial shear during forming may cause the grains to flow past each other and the semi-solid slurry will flow thixotropically. Carbides are mostly segregated to the liquid. EDS analyses at 1340°C showed that the liquid is riches in Vanadium-Tungsten-Molybdenum MC type carbides. Figure 4 (b) also indicates that delta ferrite (δ) starts to form at the temperature of 1360°C (note the δ phase surrounded by the primary austenite grain in the figure).

Liquid fraction and grain size at different temperatures (at zero minute holding) from image analysis are plotted against the liquid content from DTA in Figure 5. The liquid fraction underestimation by the image analysis, as compared to the DTA is due to insufficient cooling rate to instantaneously freeze the microstructure, resulting in further solidification of liquid on the solid phase.

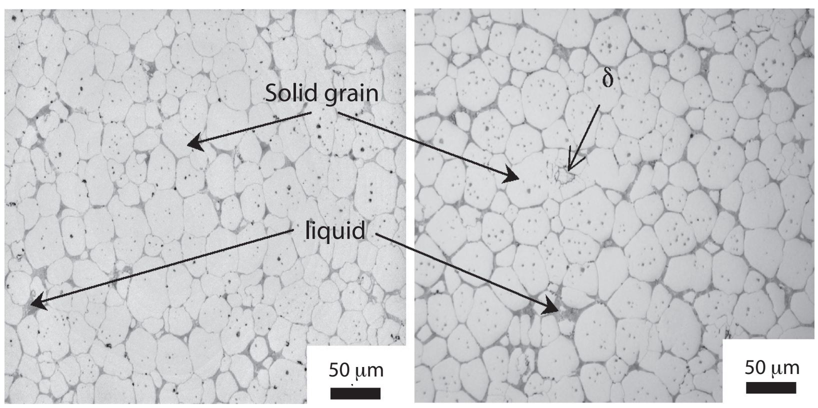

FIGURE 4. Optical micrographs of GFM M2 at (a) 1340°C and (b) 1360°C at zero minute holding.

Note that the DTA result shows that at 1320°C, the volume of liquid is 17%, while at 1340°C and 1360°C, they are 22 and 32% respectively. Grain growth is also observed whereby the mean size had increased from about 29 μm at 1320°C, to 30 and 31 μm at 1340 and 1360°C respectively.

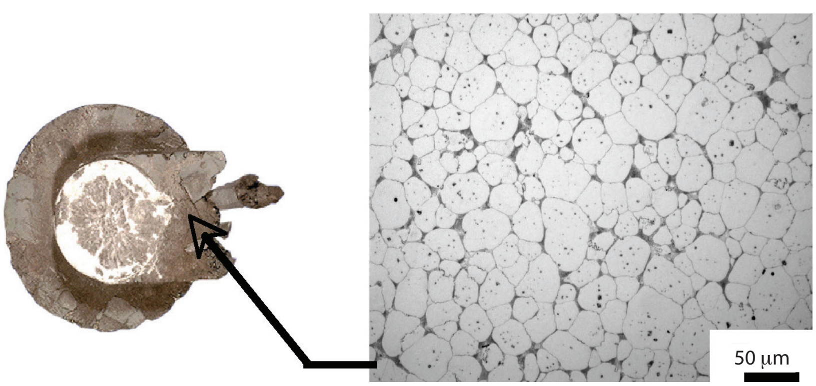

FIGURE 6. (a) An unfilled thixoformed GFM M2 demonstrator part, and (b) is the corresponding transverse structure near the die entrance at the location shown.

Thixoforming

From the results of the direct partial remelting experiments, the M2 seems to already having a suitable thixoforming microstructure at 1340°C, with a corresponding liquid fraction of about 22% (i.e. from DTA and 18% from image analysis) and grain size of 29 μm. Prior to forming, heating trials were carried out to determine suitable heating conditions (i.e. the power sequence and time) of the thixoformer induction furnace to reach the processing temperature.

The slug was heated by means of an induction system and ideally, for reasons of productivity and material characteristics, should be heated as quickly and as uniform as possible (Burke 1998 & Nohn et al. 2000). Various power settings were experimented with and it was found that a suitable heating scheme is a two stage heating with a power sequence of 24kW at the first stage and 90kW at the second stage respectively. The frequency is fixed at 1000Hz. The thixoforming press set-up at Sheffield only allows a slug to be rested on a pedestal without a holding mechanism, hence an excessive power input at initial stage of heating could cause the steel slug to jump or be displaced due to the sudden strong electromagnetic field produced by the induction system. The initial power of 24kW gave relatively rapid heating of the slug while maintaining its position inside the induction coil. The second stage at 90kW starts when the slug had reached

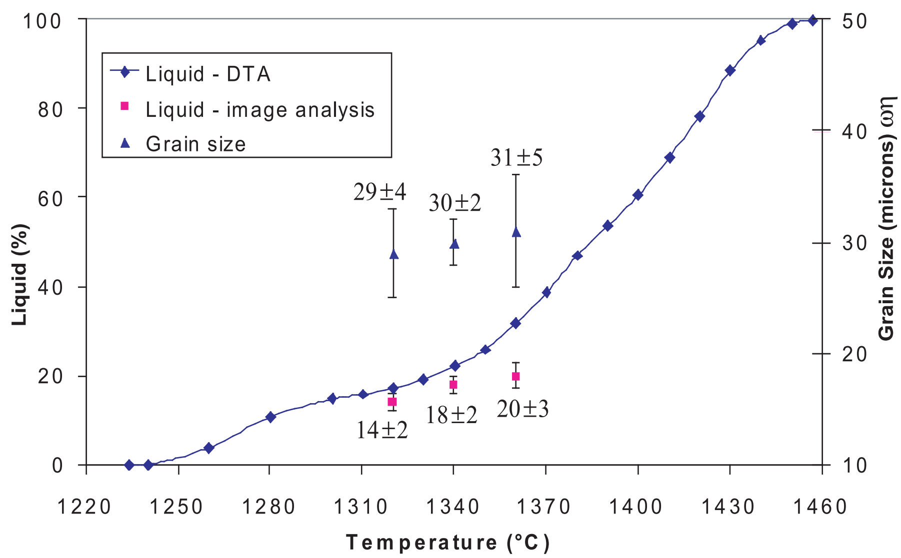

FIGURE 5. Liquid fraction and grain size from image analysis plotted with the liquid fraction from DTA.

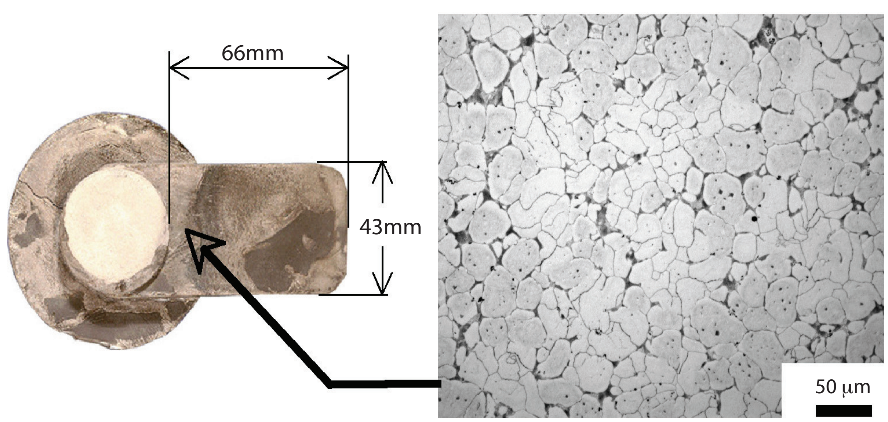

FIGURE 7. (a) A thixoformed GFM M2 demonstrator part (thickness ~7.5mm), and (b) is the corresponding transverse structure near the die entrance at the location shown.

a plateau temperature at about 780°C, its Curie point. The total heating time is approximately 8 minutes.

Figure 6(a) shows a thixoformed part forged at 500mm/s ram speed (die filling velocity), 10 seconds dwell, at the processing temperature of 1340°C. An incomplete die filling was shown to have been achieved at this processing temperature whereby the part had freeze prematurely. One reason for this is that, the low liquid content especially to sufficiently wet the grain boundaries has significantly reduced the ability of the solid grains to flow past each other during forming. The microstructure of the part near its die entrance is shown in Figure 6(b). An image analysis on this structure revealed the liquid content of approximately 17 ± 3% (compares to 18 ± 2% from the image analysis of the remelting experiment at 1340°C).

Figure 7(a) is a thixoformed part at the same processing conditions as above but at a higher processing temperature of 1360°C. A complete die filling had been achieved at this temperature with the corresponding liquid content of about 32% (i.e. by DTA). Sufficient wetting has now been achieved along the grain boundaries assisting the solid grains to flow past each other during shearing. At a glance the structure near the die entrance (Figure 7(b)) shows a small amount of liquid present. This is because liquid had mostly been pushed towards the end of the part during shearing and filling, as had also been observed by Chayong during thixoforming of aluminium 7075 (Chayong 2002). The structure also shows deformed grains morphology because of the forging pressure exerted on the compressed part and also due to the grains passing one another during flow. Figure 6 (b) on the other hand shows an equiaxed grain morphology because least pressure was experienced by the free solidification front of the unfilled part.

Figure 8 shows the load-displacement signal profiles of the two thixoformed parts mentioned above. The profiles show only part of the stroke length of the ram just after the slug first impact the top of the die at about 48mm position on the ram displacement axis. Note that the height of the slug is 46mm. The 2mm different is due to the insulating pads placed on the top and bottom of the slug to reduce heat losses. The thixoformed parts clearly exhibited two different profiles. The unfilled part (1340°C processing temperature) can be divided into four distinct zones as shown: the slug prior to it first touches the top die, zone (1); thereafter it showed a peak load at (2); followed by a significant drop at zone (3); and a rapid increase of load towards the end of the ram stroke, i.e. zone (4).

In this work, the load versus displacement curves are collected under the actual thixoforming processes. Other researchers such as Chayong (2002), Liu et al. (2003) and Hogg et al. (2004), had plotted load-displacement curves (for aluminium alloys) from rapid compression tests.

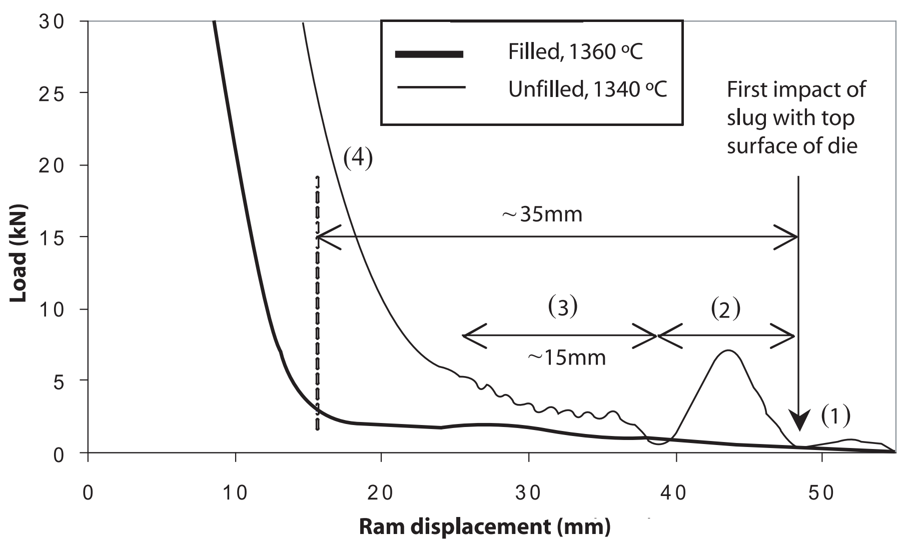

Figure 8. Load-displacement signals during thixoforming (ram speed 500mm/s, and ram movement to the left).

They carried out the tests with instrumented dies in order to simulate the thixotropic flow behaviour of materials during the rapid compression experienced by the slug during an actual thixoforming conditions. Kopp et al. (2003) on the other hand, had studied the thixotropic behaviour of a Sn-15% Pb alloy by plotting load-displacement profiles from simple compression tests. The load-displacement signals at various processing conditions from both sets of compression tests showed some similarities to the load signals from the unfilled part as shown here.

The four zones briefly mentioned above could then be explained as: (1) near zero load zone prior to reaching the die. The load is not equal to zero prior to contact with the die because in this work the load signal was collected from the ram, as against the load cell at the back of the die in Liu et al., Hogg et al. and Chayong's work. Since the load signal was converted by a pressure transducer from the hydraulic fluid pressure exerted by the ram, the movement of the ram in the majority of the stroke length will give non-zero load values prior to first impact of the billet with the die Anon (1988). (2) Structure breakdown zone. The peak load could be the initial structure breakdown load, which represents the load required for de-cohesion of a proportion of insufficiently wetted grain boundaries. The load required to overcome the resistance to shear of the broken down structure could also contribute to this peak (Hogg et al. 2004). The peak load is about 7kN. (3) Thixotropic zone. Once the breakdown phenomenon is completed, some of the solid grains are now surrounded by a liquid phase and so, achieving sufficient grain boundary decohesion; the semi-solid material now starts to show thixotropic flow behaviour. However, the liquid content of 22% is insufficient to provide enough wetting along the entire grain boundaries across the slug. As a result, this zone is shown to exhibit higher loads with a resulting shorter range (approximately 15mm flow range) as compared to its counterpart of the filled part. (4) Rapid increase in the load as the semi-solid slurry freezes (resulting in incomplete die filling).

The filled part on the other hand showed a consistently low load value during almost the entire forming operation. Note a small 'kink' at 48mm displacement, indicating a slight increase in load when the semi-solid slug first impacts the top of the die. The load during the initial shearing and breakdown zone (up to almost 35mm flow range from the first impact) is less than 2kN. The sharp increase in load at 15mm displacement is because the die approaches complete filling towards the end of the forming stroke. This suggests that at 1360°C (liquid content of 32%), the structure of the semi-solid slug should readily be broken down at the moment it touches the die, thereafter the slurry flow thixotropically successfully filling the die.

GFM M2 tool steel was successfully thixoformed at a relatively high solid content of 68% and relatively low temperature of 1360°C, which is only 25°C above its solidus and almost 100°C from its liquidus. Its microstructures are readily becoming equiaxed when reheated into the semi-solid zone without having to go through some additional cold or hot working prior to remelting as required by RAP and SIMA. The material had achieved a complete die filling when its corresponding load-displacement signals showed the absence of high structure breakdown load.

SUMMARY

The microstructures and phases of as-annealed GFM M2 tool steel in the as-received and within the semi-solid state have been studied. The as-received material shows excess carbide particles contained in bands parallel to the working direction during GFM process applied prior to heat treatment (tempering and annealing). However, when directly reheated into the semi-solid zone, the material exhibits fine equiaxed solid grains that are surrounded by liquid matrix. Most of the carbides are segregated into the liquid. The discrepancy in the volume of liquid content between the results from differential thermal analysis and those determined by image analysis is due to insufficient cooling rate to instantaneously freeze the microstructure in the latter, resulting in further solidification of liquid on the solid phase.

Thixoformings were carried out at the processing temperatures of 1340 and 1360°C, which correspond to 22 and 32% liquid respectively (from differential thermal analysis). This is because conventionally the thixoforming processing window is between 20-50% liquid. However, in the former, an incomplete die filling was achieved. Its corresponding load-displacement signals had registered a relatively high structure breakdown peak load of about 7kN, and a short thixotropic zone of about 15mm. When thixoforming was carried out at 1360°C, a complete die filling is achieved. The structure breakdown peak load is absence from its corresponding load-displacement signals. Instead, it exhibits a consistently low load value during almost the entire forming operation. This indicates that for an ideal thixoforming process of GFM M2 involving lateral flows of semi-solid slurries, the load-displacement curves should not exhibit a structure breakdown peak. Sufficient liquid is required to surround the solid grains at forging to achieve a thixotropic flow, hence achieving a complete die filling. GFM M2 tool steel can be directly thixoformed from its as-received state without having to go through additional cold or hot working prior to remelting as required by RAP and SIMA processes respectively. Thixoforming of M2 at 1360°C showed that the slurry flowed thixotropically under very low flow load of typically less than about 2 kN.

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